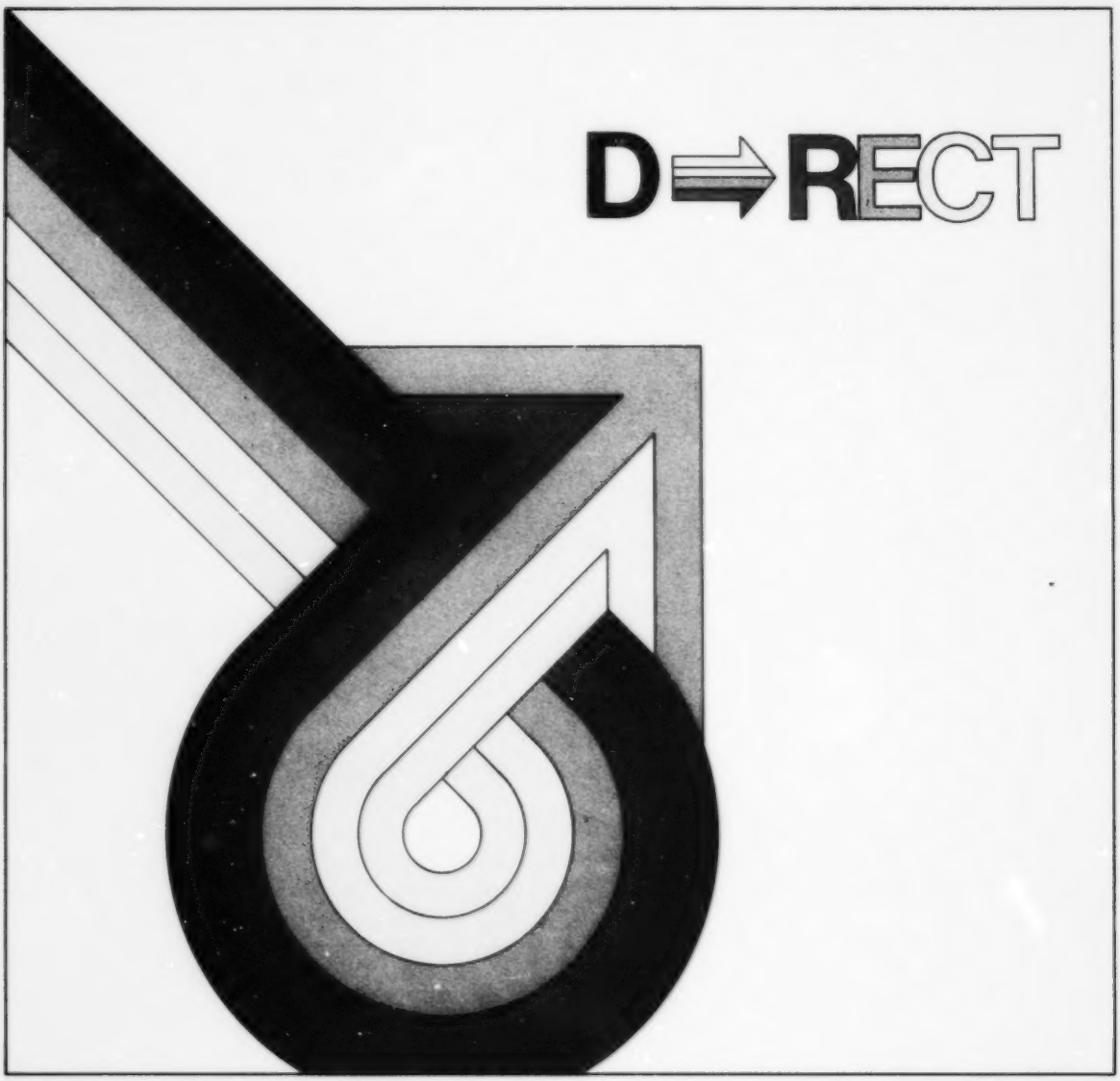
Development of a System to Combine Solvent Recovery with the Recovery of Heat from Residual Organic Wastes

Report EPS 3/CI/1
June 1989



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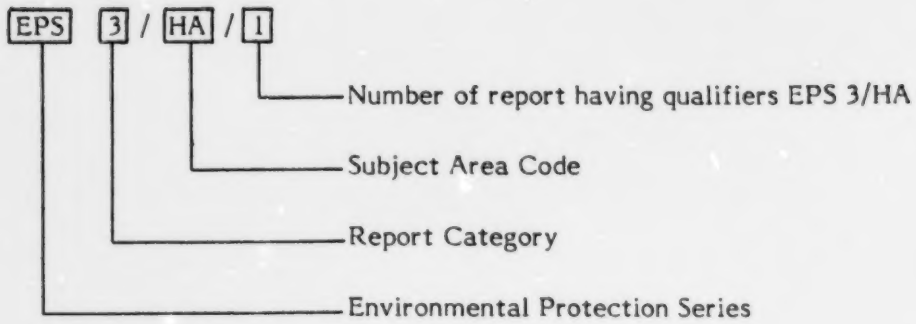
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DEVELOPMENT OF A SYSTEM TO COMBINE SOLVENT RECOVERY WITH THE RECOVERY OF HEAT FROM RESIDUAL ORGANIC WASTES

by

D.K. Jackson, W.F. Brown, K.D. Burrell and I.R. McGregor
Anachemia Solvents Limited

for the

Technology Development and Technical Services Branch
Environmental Protection
Conservation and Protection
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ABSTRACT

A process has been developed that allows the recovery of solvent and energy from still-bottom sludges produced from the conventional distillation of solvent-paint mixtures. Recovery is effected by bringing the sludge into contact with hot sand to drive off the solvent as vapour. The sand, coated with the nonvolatile portion of the sludge, is regenerated and reheated in a spouted-bed reactor. Pyrolysis gas from the reactor is sent to an afterburner where it is burned completely, the resulting heat being used to generate process steam in a waste-heat boiler. Particulate matter in the pyrolysis gas is derived from the mineral content of the sludge and it is partially removed before combustion in a set of cyclones; the balance is taken out of the flue gas from the waste-heat boiler by a baghouse.

The process is able to recover most of the solvent content of the sludge feed; the destruction of the nonvolatile combustibles produces pyrolysis gas and most of the heat required to operate the reactor. Process steam is made from combustion of the pyrolysis gas in an afterburner. The afterburner is also used to destroy solvent-bearing process water, produced by the conventional distillation of solvent mixtures.

The result of operating this process is a net economic benefit to Anachemia Solvents, its customers, and Canada.

RÉSUMÉ

On a mis a point un procédé qui permet de récupérer les solvants et l'énergie de boues résiduaires provenant de la distillation classique de mélanges de solvants et de peintures. La récupération s'effectue en mettant les boues en contact avec du sable chaud, ce qui a pour effet de chasser les solvants sous forme de vapeurs. Le sable, contenant la partie non volatile des boues, est régénéré et réchauffé dans un réacteur à lit à jet. Les gaz de pyrolyse provenant du réacteur sont envoyés à une chambre de postcombustion dont la chaleur est utilisée pour produire de la vapeur dans une chaudière de récupération. Les particules entraînées par les gaz de pyrolyse sont les minéraux des boues et elles sont partiellement extraites, avant combustion, dans un série de cyclones; le reste est extrait des gaz sortant de la chaudière de récupération par un filtre à manches.

Le processus permet de récupérer la plus grande partie des solvants contenus dans les boues. La pyrolyse des matières non volatiles produit des gaz combustibles et l'essentiel de la chaleur requise pour faire fonctionner le réacteur. On produit de la vapeur en brûlant les gaz de pyrolyse dans une chambre de postcombustion qui sert également à incinérer les eaux utilisées pendant la distillation des mélanges de solvants, qui en contiennent encore.

Le résultat de l'utilisation de ce procédé est un gain économique net pour Anachemia Solvents, ses clients et le Canada.

TABLE OF CONTENTS

		Page
ABSTRACT		iv
LIST OF FIGURES		viii
LIST OF TABLES		viii
ACKNOWLEDGEMENTS		ix
1	INTRODUCTION	1
1.1	Project Background	1
1.2	Project Process Selection	3
1.3	Environmental Considerations	4
1.4	Scope	4
2	PILOT PLANT	5
2.1	Size of the Pilot Plant	5
2.2	Process Flow	5
2.3	Pilot Plant Site	5
2.4	Blender-flasher	7
2.5	Solvent Recovery and Vapour Scrubbing	7
2.6	Reactor	8
2.7	Sand Circulation System	12
2.8	Sand Storage and Handling	12
2.9	Gas Scrubbing	13
2.10	Control Systems	14
2.11	Operation of the Process	14
2.11.1	Start-up	14
2.11.2	Self-sustaining Mode	15
2.11.3	Shutdown	15
3	DEMONSTRATION PLANT	16
3.1	Introduction	16
3.2	Blender-flasher	16
3.3	Solvent Recovery and Vapour Scrubbing	16
3.4	Reactor	17
3.5	Preheater	17
3.6	Sand Circulation System	18
3.7	Gas Cleaning	18
3.8	Incineration and Steam Generation	18
3.9	Dry Scrubber	19
3.10	Baghouse	19
3.11	Control Systems	20
3.12	Simulation Studies	21

		Page
3.13	Performance	23
3.13.1	Run 26, December 15-23, 1986	23
3.13.2	September to March Performance Summary	25
4	ENVIRONMENTAL IMPACT	27
4.1	Introduction	27
4.2	Approval by the Ontario Ministry of the Environment	27
4.3	Solids Disposal	27
4.4	Air Quality	28
4.5	Water Quality	29
5	ECONOMICS AND ENERGY SAVINGS	30
6	CONCLUSIONS AND FURTHER WORK	31
REFERENCES		32

ERRATUM

EPS 3/CI/1 July 1989

Development of a System to Combine Solvent Recovery
With the Recovery of Heat from Residual Organic Wastes

Page 25, Table 2 total tonnes under Heat Sources should read 54 not 52

LIST OF FIGURES

Figure		Page
1	TYPICAL "DIRTY SOLVENT" COMPOSITION	2
2	TYPICAL PAINT SLUDGE COMPOSITION	2
3	VOLATILATOR PROCESS FLOW DIAGRAM	6
4	PILOT PLANT SITE PLAN	6
5	BLENDER-FLASHER	8
6	SOLVENT RECOVERY AND VAPOUR SCRUBBING SYSTEM	9
7	REACTOR	10
8	SAND CIRCULATION SYSTEM	13
9	EFFECT OF PROCESS WATER INJECTION INTO THE AFTERBURNER	22

LIST OF TABLES

Table		
1	MATERIAL BALANCE FOR RUN 26 (Dec. 15-23, 1986)	24
2	HEAT BALANCE FOR RUN 26 (Dec. 15-23, 1986)	25
3	FLUE GAS IMPINGEMENT CONCENTRATIONS (half-hour average)	29

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1 INTRODUCTION

1.1 Project Background

Manufacturers who make paint or who paint products in a variety of colours must clean their equipment whenever they change the colour. In the automotive industry, in particular, routine production entails frequent colour changes, which require flushing the equipment and lines with solvent. As a result, substantial volumes of dilute paint of nondescript colour are produced. There are also many other manufacturing processes that produce contaminated solvents. These materials must not be sent to normal municipal waste disposal sites, but must be reclaimed or incinerated so that they do not become a burden on the municipal effluent system and the environment. The reclaimed solvent is a source of cheap wash solvent. Solvent recovery has a second effect on the general economy, i.e., less crude oil is required to manufacture virgin solvent, thus conserving resources or reducing import dollars. Solvent recycling companies provide this service, without which the solvent users would probably have to set up their own reclamation operations without the volumes necessary to make the operations profitable.

Anachemia Solvents, located just west of Toronto in Mississauga, is in the business of reclaiming such solvent and returning it to its customers. Anachemia's operation, therefore, has a direct beneficial impact on the environment and on its customers' solvent costs. It is the largest solvent recycler in Canada.

Anachemia Solvents uses conventional batch stills and falling-film evaporator stills to separate solvent from contaminant material. Solvent is removed until the temperature of the residue or its chemical nature make further recovery a risk. (Some paints contain resins which cure rapidly when hot and exposed to air or some other curing agent. Attempts to recover additional solvent by conventional means entail substantial risk of polymerizing the resins and converting the residue into a solid mass.) The composition of a typical "dirty solvent" is shown in Figure 1. Conventional distillation separates this dirty solvent into a distillate with a solvent phase and a water phase, and still-bottoms, often referred to as sludge or paint sludge. The paint sludge contains about 50% solvent, 25 to 40% resins, 5 to 20% water, and 5 to 10% solids, as shown in Figure 2.

Anachemia Solvents has the problem of disposing of this sludge. Historically, it was simply incinerated on-site. Some of the heat from this incineration was used to make steam, the remainder being discharged to the atmosphere unused. Some paint solids were deposited as ash in the incinerator and were hauled away to a municipal waste site.

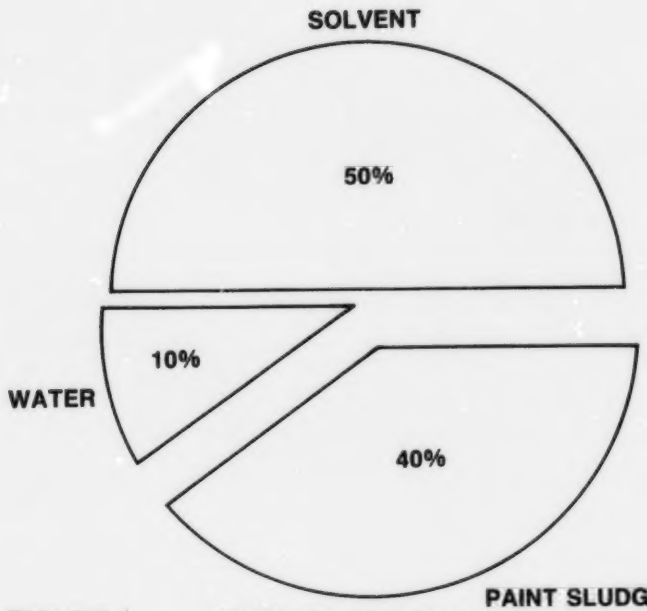


FIGURE 1 TYPICAL "DIRTY SOLVENT" COMPOSITION

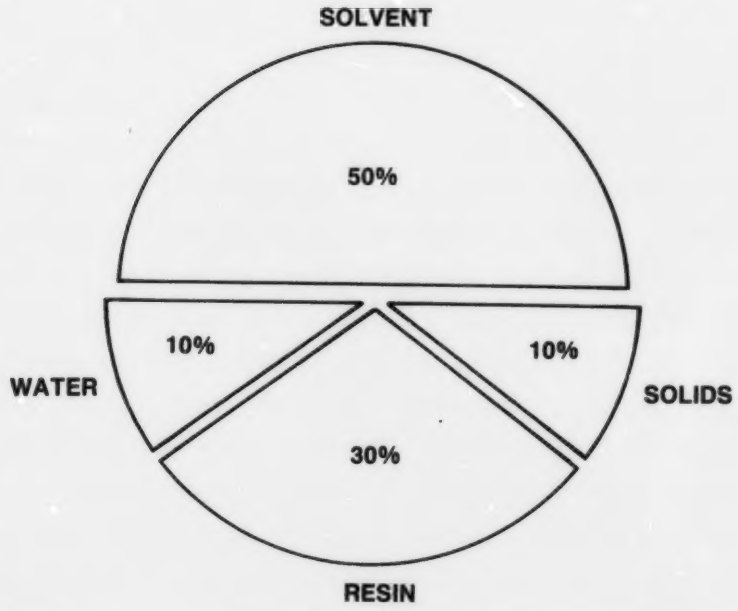


FIGURE 2 TYPICAL PAINT SLUDGE COMPOSITION

In those cases where production of still-bottom sludge exceeded incinerator capacity, the excess had to be hauled away to a secure landfill or another incinerator. These off-site disposal sites were frequently in the U.S.

Hauling excess sludge to a disposal site is increasingly expensive (current disposal costs are about 10 cents/litre); moreover, the amount of sludge that can be shipped is limited by the number of available registered sites. At times, all sites had more business than they could handle, and none of the sites was in Canada. From the above, it can be seen that it is in Anachemia's interest to be able to process, on its own site, as much of the waste from its recycling operations as possible. However, there are significant problems associated with incineration of the sludge. The incinerator stack gas contains a high concentration of particulates. In November 1982, Anachemia's stack-gas emissions were monitored by the Ontario Ministry of the Environment (MOE) and were found not to be in compliance with environmental regulations. Approximately 65% of the paint solids in the sludge were emitted with the stack gas, the remainder being deposited in the incinerator, which needed to be cleaned out weekly. In June 1984, a Control Order was issued requiring Anachemia Solvents to modify the incinerator so that it could be operated without emitting excessive particulates, lead, titanium, or hydrogen chloride, or to shut down the incinerator by September 1984. Any new facility would require a Certificate of Approval from MOE. (Conditions required for approval were set out in a letter from MOE in May 1986.) Appeal procedures probably could have delayed implementation of the Control Order, but having been made aware of the problem, Anachemia chose instead to seek an engineering solution immediately.

1.2 Project Process Selection

Several process options were examined by W.F.M. Brown Associates, including the simple addition of a lime scrubber to remove acid gases from the flue gas and a baghouse to trap particulate matter. Most of the options examined constituted a continuing extra expense without a payback. However, one option did offer the promise of a payback; it was a process to recover additional solvent from the still-bottoms and energy from burning the resin fraction, and to trap the particulate matter in a baghouse. Recovery of additional solvent promised to provide additional revenue, reduce the heat wasted, and reduce the volume of flue gas, thereby reducing the size of the baghouse and lime scrubber. To recover the additional solvent, however, a method had to be devised to keep the residue in a form that could be moved, and not to allow it to form a solid mass in the process vessel. The essential concept was to use hot sand as a source of heat to drive

off solvent, and to use the surface of the sand as an acceptor for the residue. The coated sand would be transferred to a reactor where substoichiometric combustion would destroy the residue and heat the sand for reuse. For the reactor, a spouted-bed design was chosen. A fluid-bed reactor would also be effective, but a spouted bed[1] was selected because it requires about 40% less air (and, therefore, blower power) to operate it.

1.3 Environmental Considerations

The new process[2] would have several effects on the environment. Like any process meeting the requirements of the Control Order, it would allow continuation of the solvent-recovery operation, thus avoiding imposing on the environment the burden of unrecovered solvent (e.g., secure landfill sites for the unprocessed dirty solvent and additional crude processed to replace solvent that is not reclaimed). If the requirements of the Control Order could be satisfied by simply adding a baghouse to the existing facility, the unrecovered solvent in the sludge would still be a loss, and the heat liberated by its combustion would have to be extracted from the flue gas to protect the baghouse, even though it would be too much to be absorbed in other processes at Anachemia. Only the new process offered the promise of virtually complete recovery of solvent and solids, thereby imposing a minimum burden on the environment.

1.4 Scope

This report describes the development of the concept, the structure of the plant and its operation, and performance data for the demonstration plant.

2 PILOT PLANT

2.1 Size of the Pilot Plant

At the beginning of the project, it was decided that a pilot plant should be built at commercial scale, not at bench scale. This decision was taken because, at that time, available scale-up correlations appeared to be completely unsatisfactory. Therefore, the pilot plant was built as cheaply as possible, without undue regard for the useful life of components, as there was a high risk that a given component would be found to be unsatisfactory and would need to be replaced with something different. Components that were found to be satisfactory would be replaced with sturdier components when necessary.

2.2 Process Flow

Process flow is shown as a block diagram in Figure 3 ("VOLATILATOR" is the trade name of the process). Feed (still-bottom sludge) and hot sand were introduced into the blender-flasher separately and were mixed. The heat in the sand flashed the solvent contained in the sludge and produced some incidental cracking if the sand was hot enough. Solvent vapour went to solvent recovery; whereas the dirty sand, coated with the nonvolatile part of the feed, was sent to the reactor. The reactor was operated under substoichiometric (starved-air) conditions. In this mode, the resin fraction was partially combusted and partially cracked, thereby regenerating the sand. The products of the reactions were pyrolysis gas, coke, mineral ash, and enough heat to restore the sand temperature for reuse in the blender-flasher. As noted above, a stream of regenerated sand was sent back to the blender-flasher. Pyrolysis gas, which contained solids, was passed through cyclones to separate the solids, and was then sent to the afterburner where it was burned.

2.3 Pilot Plant Site

The pilot plant (Figure 4), located beside the existing steam plant, occupied an area of approximately 12 x 15 m. A trailer for control, storage, and administration was also located on the pilot plant site. The pilot plant consisted of a blender-flasher, a condenser, a reactor and cyclone, a sand storage vessel, and associated piping. The pilot plant was tied into the existing incinerator for burning the pyrolysis gas.

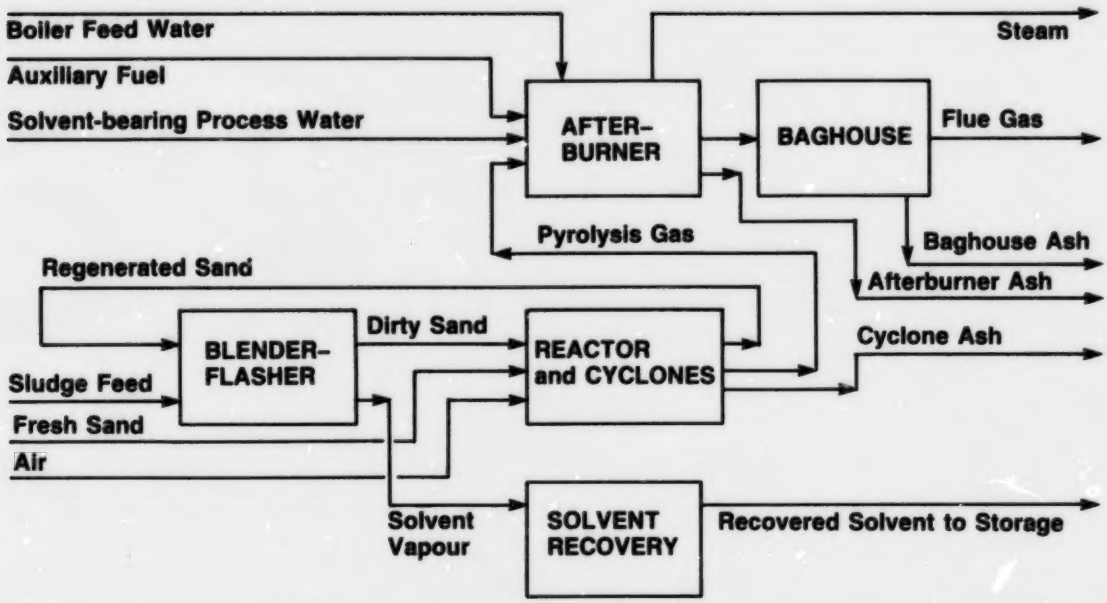


FIGURE 3 VOLATILATOR PROCESS FLOW DIAGRAM

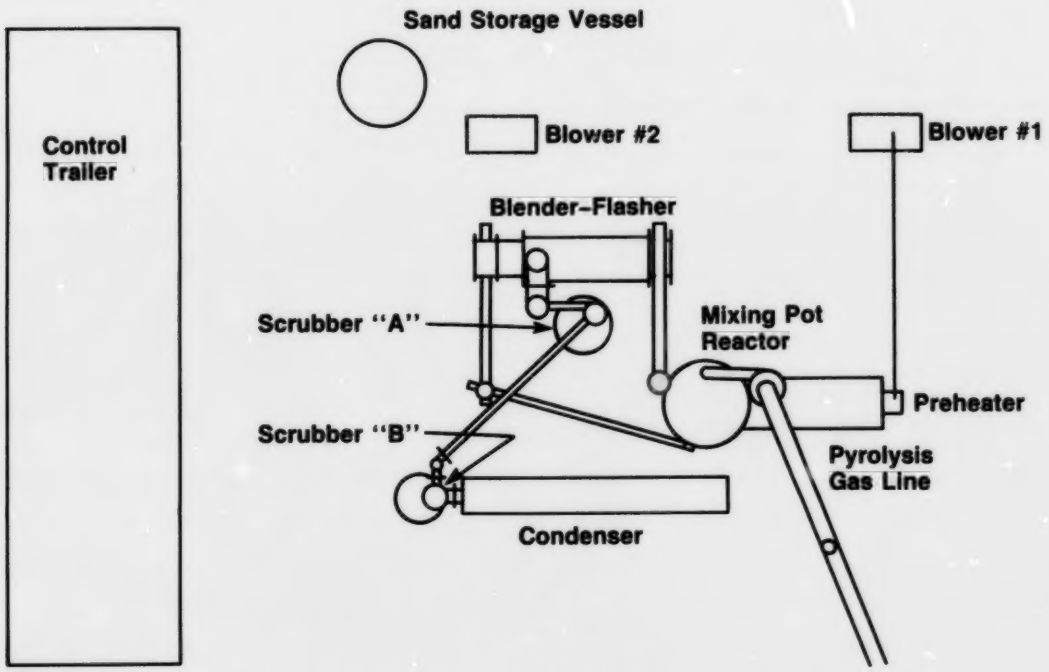


FIGURE 4 PILOT PLANT SITE PLAN

2.4 Blender-flasher

The blender-flasher is illustrated in Figure 5. Its function was to separate sludge into its volatile (solvent and water) and nonvolatile (resins and minerals) components by bringing the sludge into contact with hot sand. The blender-flasher consisted of two horizontal cylinders (drums) mounted one above the other, attached to form a closed vessel. The lower drum was the blender and the upper drum was the flash chamber.

The blender was 500 mm in diameter and 2400 mm in length. At the intake end, it was fitted with a 150-mm diameter horizontal pipe mounted transversely above the blender cylinder, through which a screw ("regen. sand screw") delivered regenerated sand. Sludge was introduced into the blender-flasher with a positive displacement pump which drew its feed from the existing sludge incinerator sludge feed system. The pump, driven by a 0.4-kW, 90-V DC motor, sent sludge through a flexible steel line to an oscillating sludge nozzle, which dropped the sludge along the bed. Since sludge forms hard deposits on hot undisturbed surfaces, it was essential to disperse the sludge quickly in the sand.

The blender was equipped with mixing paddles mounted on a 70-mm shaft which turned at about 20 r/min. These paddles mixed the sludge with the hot sand from the reactor, thereby heating the sludge and flashing the solvent. The nonvolatile portion of the sludge remained on the sand as a coating. This mixture, called dirty sand, was pushed through the blender and over a semicircular dam by the mixing paddles into an exit chamber. The dam kept the sand up to the level of the shaft. Sand was pushed over the dam and fell through an exit cone onto the lower dirty-sand screw. The flashed vapour rose into the flash drum where it was ducted by a 250-mm riser and a 100-mm diameter line to the vapour scrubber system and then to the condenser.

2.5 Solvent Recovery and Vapour Scrubbing

The vapour flashed off the sludge by the hot sand contained coke and other solids as well as solvent and water vapours. Some of the resin fraction cracked, forming coke particles which had to be scrubbed out of the solvent vapour. These particles were scrubbed from the vapour by a recirculation system which used a portion of the solvent product. The system is illustrated in Figure 6. In the first scrubber (scrubber A), vapour was brought into contact with scrubbing liquid at a wetted-wall cone followed by a venturi restriction. The liquid/vapour mixture was disengaged in a tower, with the liquid draining to the loop A settler and the vapour proceeding to a second scrubbing tower (scrubber B).

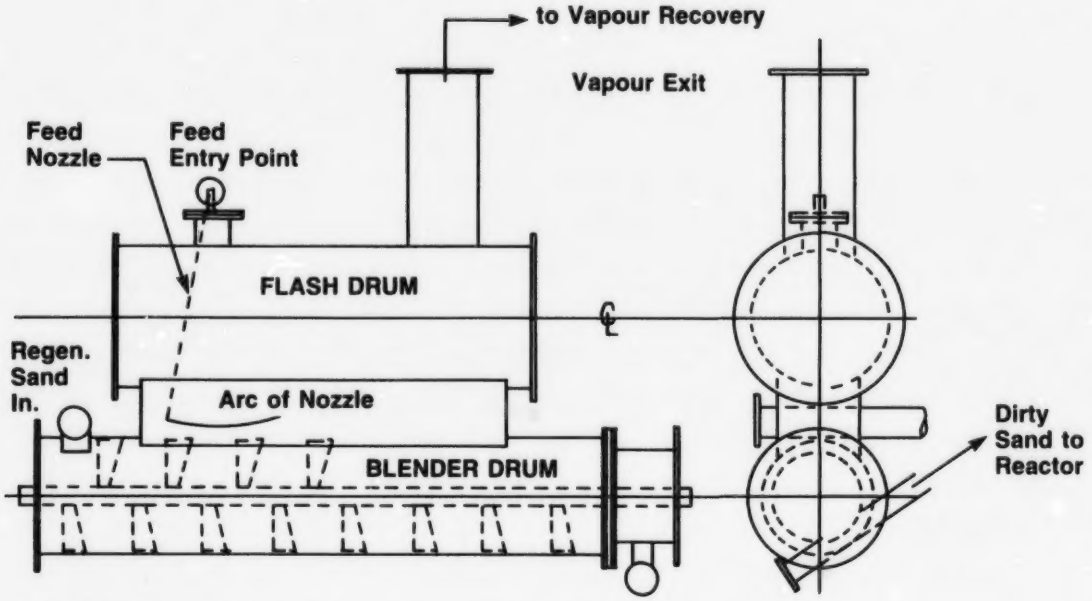


FIGURE 5 BLENDER-FLASHER

Solid material removed from the vapour in scrubber A was discharged as a slurry to the sludge feed vessel, and was recycled to the flash chamber. Under this procedure, solids were eventually carried out with the dirty sand while the solvent was reflashed.

The second scrubber also had a circulating liquid, supplied from the solvent product. Solids collected in this loop were discharged to the solvent product; this was acceptable because the solvent was reprocessed or burned. The circulating solvent for the second scrubber also supplied solvent makeup to scrubber A.

2.6 Reactor

The reactor had three functions: to remove sludge residue from the sand, to reheat the sand to act as a heat carrier to the flasher, and to generate pyrolysis gas for the afterburner and heat-recovery sections. The reactor section consisted of the reactor and a preheater furnace.

The reactor vessel (Figure 7) was a steel cylinder, 1200 mm in diameter by 2750 mm high, with a conical bottom, 810 mm deep, terminating in a 200-mm flange. The top of the reactor consisted of a flat plate with a 500-mm manhole flange and a gas exit duct made of 200-mm diameter pipe. The reactor was supported on legs which raised the cylinder/cone intersection 2.1 m above grade. The outside of the reactor was insulated with preformed magnesia slabs. The reactor internals consisted of a draft tube, several

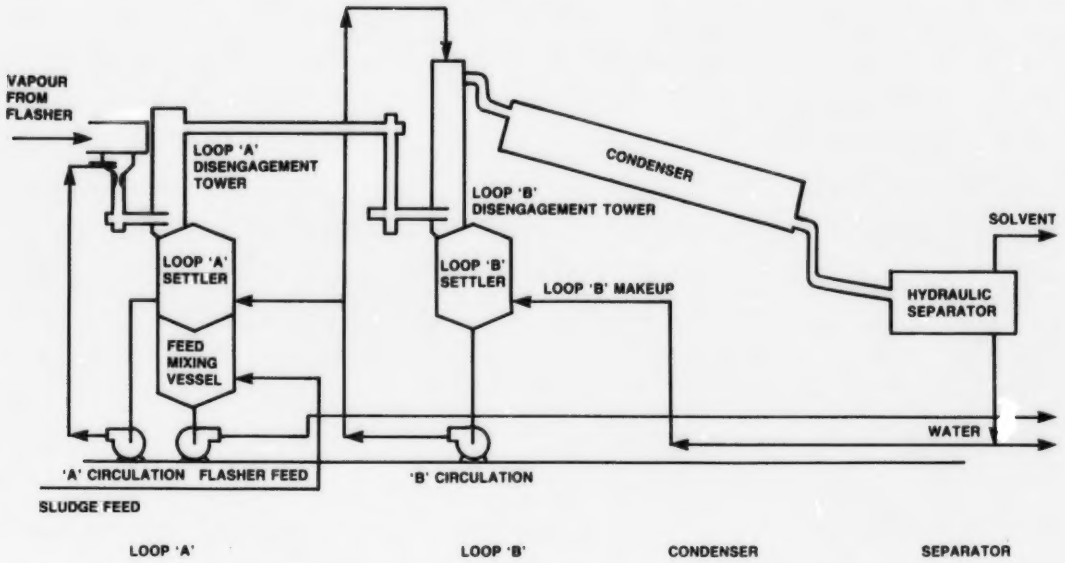


FIGURE 6 SOLVENT RECOVERY AND VAPOUR SCRUBBING SYSTEM

thermocouples, a refractory lining, a supplementary air distributor, and a dirty-sand inlet. The draft tube was a vertical cylinder, 200 mm in diameter by 700 mm high, mounted concentrically 300 mm above the bottom of the reactor. It directed hot air from the preheater through the bed to aid the spouting action.

Seven thermocouples were installed in the reactor as follows: at the base of the spout, about 150 mm above the throat; at the north annulus (3), approximately at the junction of the cylinder and cone, on the north side, at varying radial distances; at the south annulus, opposite the north annulus thermocouples; at the top of the bed, on the north side, about 300 mm above the north annulus thermocouples; and in the freeboard 600 mm below the pyrolysis gas exit.

Primary air to the reactor was supplied by a rotary-lobe blower with a capacity of approximately 80 L/s. This air was routed through the preheater into the reactor to provide the spouting action. The air was heated by the preheater burner when the reactor was not able to generate its own heat, e.g., during start-up or when the bed contained insufficient fuel. Additional air was supplied to the air distributor from blower No. 2 to control the extent of combustion in the reactor.

The preheater was a horizontal steel cylinder 2200 mm long and 700 mm in diameter. The burner end of the preheater cylinder was fitted with a flanged end plate

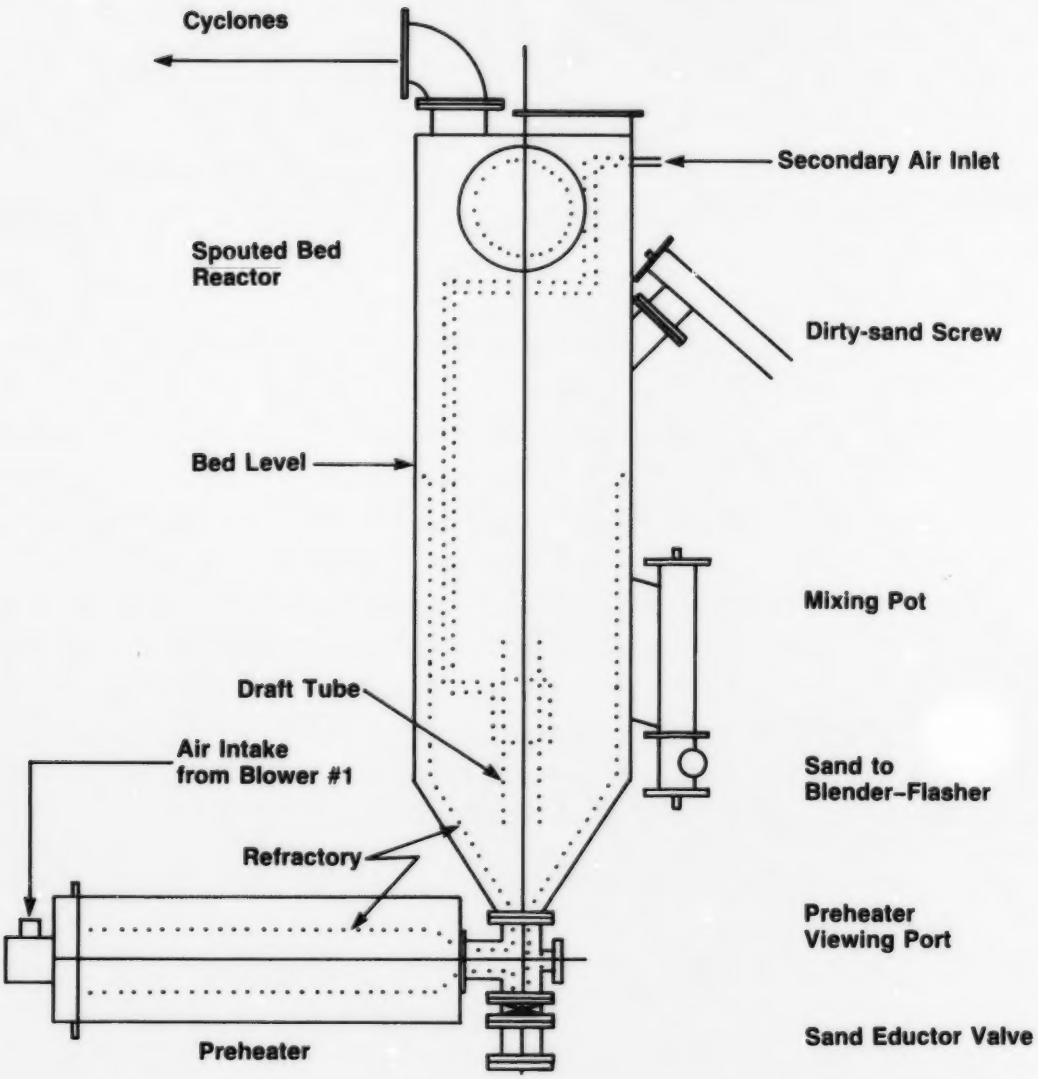


FIGURE 7 REACTOR

upon which the burner was mounted. The other end was closed by a flange of the same diameter as the preheater cylinder. This flange was fitted with a 100-mm, air-cooled viewing port. The preheater cylinder was insulated with 180 mm of refractories, leaving a 350-mm cylinder to conduct heated air to the reactor. A nozzle was located 300 mm from the downstream end to conduct gases into the reactor. A second nozzle was located on the underside of the preheater to allow for the removal of sand which fell into the preheater from the reactor. In this region, the refractory had been shaped to allow sand to drain out. The lower nozzle was fitted with a venturi structure and shutoff valves to move sand out of the preheater to the sand storage hopper.

The preheater burner was a TEMPEST II dual-fuel high intensity burner, chosen because it is capable of operating above ambient pressure. It had a capacity of 150 kW.

During operation, dirty sand was received from the flasher via the dirty-sand screws. The sand formed a bed in the reactor. The hot air from the preheater jetted up through the bed, bringing with it entrained sand, forming a spout or fountain; this device constituted the spouted bed. The spout provided intimate contact between the sand particles and the preheater exit gas so that they were brought rapidly to thermal and chemical equilibrium. The sand fell back onto the bed and a portion flowed out of the reactor by gravity into the mixing pot via a slotted opening 600 mm high by 200 mm wide. Sand that did not return immediately to the flasher moved down through the annulus until it was again injected into the spout and fell onto the top of the bed. The pyrolysis gas exited from the top of the reactor to a cyclone separator.

Preliminary removal of paint solids, char, and sand fines was accomplished by the cyclone separator. This mixture is referred to as ash. Attached to the cyclone separator were a dust collection chamber, an ash removal screw, and a drum loading facility. The cyclone was of conventional design, 400 mm in diameter. A 100-L dust settling chamber was attached to the bottom of the cyclone. The settled dust was released from the settling chamber to an ash removal screw, which routed the solids to a drum at grade level. The drum was placed on a roller conveyor to facilitate changing drums.

The pyrolysis gas from the cyclone was led through an insulated, 200-mm steel line to the present sludge incinerator where it was discharged into the incinerator firebox at three points in a plane 1200 mm from the inlet end of the firebox.

2.7 Sand Circulation System

Sand circulation was an integral part of the process. Hot sand had to be brought into the flasher to act as a heat carrier to flash solvent from the sludge. The sand also had to act as a resin/paint-solids carrier to bring the nonvolatile portion of the sludge into the reactor where it could be burned to provide heat and pyrolysis gas. These functions had to be accomplished with a minimum exchange of gases between the reactor and flasher to avoid product loss and product contamination. The sand circulation system (Figure 8) comprised a mixing pot attached to the reactor, a screw to bring regenerated sand from the reactor to the flasher via the mixing pot, and two dirty-sand screws in series to carry the dirty sand back to the reactor.

The mixing pot was a cylinder equipped with vertical stirring bars. The stirring bars were driven by a 0.75-kW, 90-V DC motor. The current drawn by the motor provided an indication of the sand level in the mixer and, hence, in the bed, and was the basic tool for measuring bed depth. The rate at which sand dropped from the mixing pot into the regen. sand screw was governed by the speed of the screw.

The regen. sand screw ran from the bottom of the mixing pot to the blender-flasher and transported sand from the mixing pot to the blender-flasher. The regen. sand screw was a 100-mm screw; 2000 mm long; driven by a 0.75-kW, 90-V DC motor; and housed in a 125-mm diameter pipe.

The dirty-sand screws carried the dirty sand from the blender-flasher to enter the reactor at a height of 3.6 m above grade. The lift was accomplished in two stages (Figure 8) so that standard length screws could be used, and to avoid steep angles at which screws are very inefficient. The screws were mounted 40° from the horizontal inside 150-mm diameter pipes. Each of the screws was 150 mm in diameter by 2100 mm long, driven by 1.5-kW, 110-V AC motors. Originally, the motors drove the screws through hollow-shaft gearboxes; however, these failed and jammed, and were replaced by a chain drive and a solid-shaft gearbox. Between the two dirty-sand screws, a vertical cylinder 200 mm in diameter by 600 mm long served to transfer sand from the lower screw to the upper screw. Sand was delivered into the reactor by the upper sand screw via an inclined pipe.

2.8 Sand Storage and Handling

The supplementary air blower (blower No. 2, Figure 4), which provided supplementary air to the reactor during the run, also provided air to move sand pneumatically from the reactor to the sand storage vessel and back.

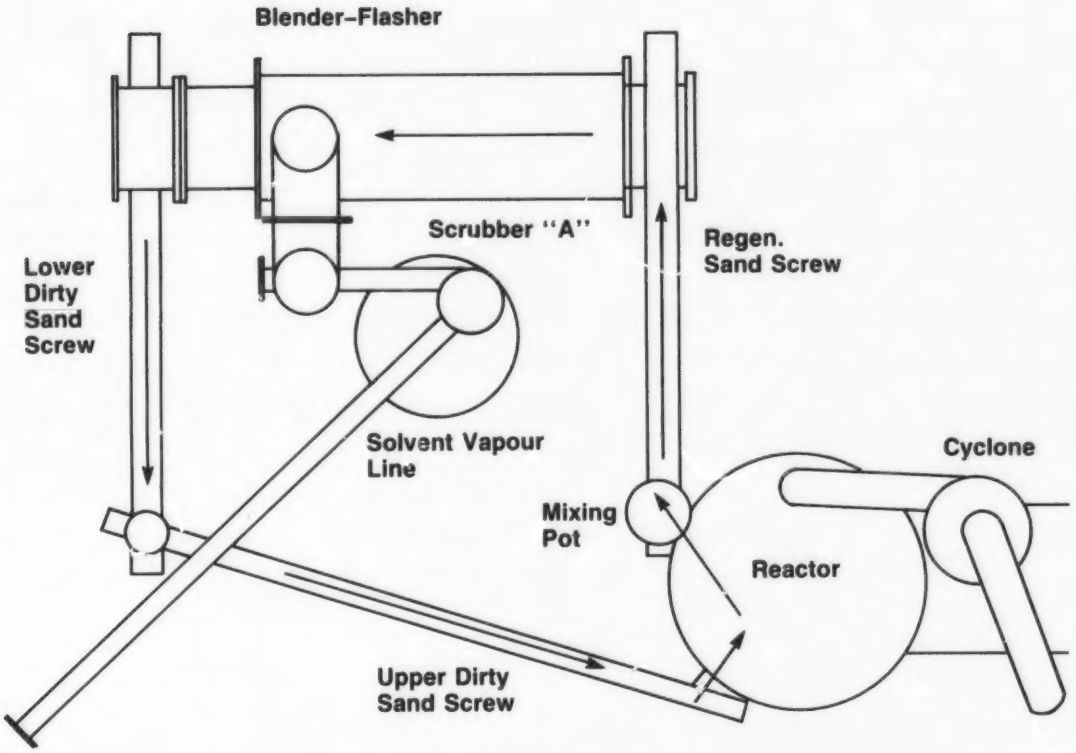


FIGURE 8 SAND CIRCULATION SYSTEM

The sand storage vessel was a cone-bottomed cylindrical tank mounted in a structural steel frame. Under the cone was a sand addition hopper fitted with a venturi and necessary lines to blow sand to the reactor. Sand was moved from the reactor by a similar venturi located under the reactor preheater. Motive air for both venturis was supplied by the supplementary air blower.

2.9 Gas Scrubbing

Pyrolysis gas from the reactor was a hot mixture of nitrogen, fuel components, and particulate matter. The latter consisted of ash with about 20% coke and tars. The bulk of the particulate matter was removed by a cyclone separator, and the separated solids were led to an ash drum through a screw and a shutoff valve. However, it was found that the pyrolysis gas line from the cyclone to the afterburner was prone to

plugging with solids. Therefore, a Ducon wet-gas scrubber was proposed for the demonstration plant to clean the flue gas.

2.10 Control Systems

Process controls were located in a 5-m trailer, which also served as the entry point for power and air utilities. All process temperatures were monitored using a Radio Shack TRS 80 Model I computer with one disk drive and a printer. These temperatures were displayed on the computer screen, and a selection of them was printed on a time-temperature graph to show trends. The operation of screws and pumps was monitored by means of the current drawn by their respective motors. Reactor and flash-chamber pressures were monitored in the control trailer by manometers. Preheater pressures were monitored directly by pressure gauges. Airflow from the preheater blower was not measured because the blower was run at capacity; airflow directed to the air-distributor system in the reactor was inferred from the pressure downstream from the corresponding control valve, shown on a gauge mounted in the trailer. The control valve was adjusted as necessary to control reactor temperatures. Product inventory was displayed in the control trailer.

2.11 Operation of the Process

2.11.1 Start-up. The start-up proceeded in three well-defined phases. First, the spouted-bed operation was established:

- Afterburner operation was established so that any gas formed in the reactor would be burned safely.
- Sand was added to the reactor in quantities sufficient to maintain inventory. This amounted to about 3 t total inventory.
- Air blower No. 1 was started, directing air into the reactor through the preheater. This established spouting in the reactor.

Second, the reactor was heated to operating temperature. The preheater was ignited to heat the sand with hot air. When the sand temperature reached 425°C, sand circulation was started to warm the sand screws and flasher to operating temperature.

The third phase was the period from the introduction of feed until the process could operate without burning fuel in the preheater. After reaching operating temperature, the vapour scrubbers' circulations were started, and feed was brought into the blender-flasher. The process was operated in this mode until sufficient fuel existed in

the reactor bed (as uncombusted cracked resin and char) to maintain combustion within the bed. Blower No. 2 was used to introduce supplementary air into the reactor. If the reactor temperature rose, this indicated that the bed temperature and fuel content (resin and char) were sufficient to maintain combustion in the bed. Fuel to the preheater burner was stopped, thus providing more free oxygen to the reactor. The sludge feed rate to the blender-flasher was adjusted to absorb the additional heat generated and to provide more fuel to the bed. Thereafter, the secondary air rate and the sludge feed rate were adjusted to maintain reactor temperature within the required operating range. The process was then said to be in a self-sustaining mode because combustion of char in the reactor released enough heat to sustain cracking reactions to form char and to flash solvent out of the feed in the blender-flasher.

2.11.2 Self-sustaining Mode. The self-sustaining mode was the desired mode of operation of the process. Temperatures in the reactor and blender-flasher were important indicators of the state of the process. In the blender-flasher, sand was cooled by admixture with the feed; the drop in sand temperature was a direct indication of the adequacy of the ratio of sand to feed. The sand exit temperature was an indication of the completeness of solvent removal from the sludge. In the reactor, the temperatures indicated the state of the combustion and regeneration processes, e.g., if an increase in the secondary airflow rate led to cooling in the bed, this indicated that free oxygen was available and that the fuel content in the bed might be too lean to sustain the process. In such a case, auxiliary heat from the preheater would be required until the fuel content of the bed was restored and the self-sustaining mode was resumed. As long as the bed was rich in fuel, the temperature could be controlled by the air-addition rate. As a result of the variable composition of the sludge, such response patterns are more reliable than any calculated values. The feed rate is maximized subject to temperature drop through the blender and the ability to maintain adequate reactor temperatures.

2.11.3 Shutdown. The process needs to be shut down at the end of the work week, and at other times for maintenance or because of abnormal conditions. The shutdown process, in many ways, was the reversal of start-up. The feed was stopped, and auxiliary air to the reactor was discontinued. Sand circulation was continued until only regenerated sand was contained in the circulation system. Blower No. 1 was shut off. Auxiliary systems, such as cooling water, were shut off as their function became unnecessary. Sand was left in the reactor if possible.

3 DEMONSTRATION PLANT

3.1 Introduction

The demonstration plant was constructed by converting the pilot plant. This was possible because, as noted previously, the pilot plant had been built at commercial scale. The existing incinerator was changed to function exclusively as an afterburner, and a baghouse was added to trap particulate matter in the flue gas. At that time, as well, there was a distinct possibility that Anachemia's entire solvent-recovery operation might be moved to a new location. This led us to keep any part of the pilot plant that could serve in the demonstration unit. Conversion consisted of upgrading successful features, revising some equipment, and keeping other equipment. Many changes have been made to the pilot plant since it first produced solvent in May 1984, usually in response to operating or maintenance problems. In addition, disposal of solvent-bearing wastewater by injecting it into the afterburner firebox became an important part of our effluent-water-quality control program. The solvent content of such waters can range from 20 000 mg/L COD (chemical oxygen demand) to 20% by weight. These waters are said to be "burned" in the afterburner. Indeed, some of them contain enough fuel value to heat the water and the products of their combustion to flue-gas temperatures. Nevertheless, the essential process has remained as shown in Figure 3. The following discussion outlines changes to March 1987.

3.2 Blender-flasher

The blender-flasher was replaced with a similar unit which had a greater wall thickness in the blender. The blender paddle-shaft stuffing boxes were replaced with steam-purged lantern rings to keep solvent vapour from escaping. The sand-exit structure was redesigned. Instead of having the sand drop freely through a cone to the dirty-sand screw, the screw was raised to intersect the blender cylinder in a special exit drum. The exit drum was fitted with a set of paddles shaped to scrape the wall without interfering with the dirty-sand screw and to push the dirty sand directly into the dirty-sand screw.

3.3 Solvent Recovery and Vapour Scrubbing

The flashed vapour from the blender-flasher goes to two scrubbers in series which remove entrained particulate matter from the solvent vapour, and return it to the flasher with the fresh feed. A high-boiling-point liquid derived from the high-boiling-point components of the vapour is jetted cyclonically around a short cone

fitted with a pinch valve at its lower end. The pinch valve provides intimate contact between the solvent vapour and the scrubbing liquid and delivers the mixture to a vapour/liquid separator. The vapour/liquid separator is fitted with an automatic valve which delivers a small volume of scrubbing liquid to a tank below, which contains the fresh feed. By this means, solids scrubbed from the vapour are returned to the flash chamber with the feed and are sent out with the dirty sand. The second scrubber consists of a tower filled with ceramic saddles and wetted by recirculating solvent. After this scrubber, the vapour goes to a shell-and-tube condenser. The condensate, consisting of water and solvent, is pumped to storage. A hydraulic separator, initially used to separate these components, was removed from the process because it was found that it was easier to allow separation to take place in storage.

3.4 Reactor

The height of the reactor was increased by 760 mm, and the roof was replaced as it had been subjected to sandblasting by the sand jet. A baffle was added at the outlet to the cyclone to reduce the carryover of particulate matter. The internal refractory was renewed. The insulation installed outside the reactor was changed to 50-mm Kaowool and metal lath, covered with asphaltic waterproofing. Access to the reactor shell for maintenance or new installation can be gained with only local damage being sustained.

3.5 Preheater

Sand in the spouted bed always flows down into the preheater firebox when the primary blower is shut off. The conical exit used in the pilot plant to remove sand was not completely satisfactory because sand continued to be left in the preheater firebox and to restrict air intake. To correct this problem, a new firebox and a new breeching to the reactor were built. The new breeching provides a narrower horizontal path, which better contains the sand within the region where it can be conveyed pneumatically to storage. A Lowe valve, which had been the shutoff to the sand line to storage, has been replaced with a knife valve. The revised facility works well.

The operating temperature in the firebox can be very high; during one run, for example, the refractory was destroyed. The new firebox, therefore, was relined with two layers of refractory, the innermost layer being a special grade of high-temperature material and the second layer having a lower service temperature limit but a better insulating value than the inner material.

3.6 Sand Circulation System

Gearbox failure in the dirty-sand screws has been a problem, which is thought to be due to grit carried into the gearbox seal. The gearboxes have been replaced with chain drives, which have proved more reliable.

3.7 Gas Cleaning

The removal of particulate matter from the pyrolysis gas is accomplished by cyclones in series after the reactor. Their function is to remove mineral solids and coke and to pass the cleaned gas to the afterburner for combustion. However, even after passing through the cyclones, the pyrolysis gas still contained coke and high-boiling-point liquids which were deposited in the line to the afterburner. A Ducon-Micropul wet scrubber was installed in the pyrolysis gas line between the reactor cyclone and the afterburner. It removed particulate matter, but it also upset the pressure balance around the cyclones; thus, the wet scrubber had to remove all the particulate matter from the pyrolysis gas. This load was too great and the wet scrubber had to be abandoned. A more effective remedy was to insulate the pyrolysis gas line.

3.8 Incineration and Steam Generation

Pyrolysis gas must be incinerated completely and the resulting flue gas must not be released to the atmosphere unless it meets the requirements of the MOE Control Order (June 4, 1984) and the conditions for the Certificate of Approval (May 5, 1986). These requirements are discussed in Section 4. Simulation studies were conducted indicating that the existing sludge incinerator could be adapted to become the afterburner. The incinerator was a horizontal cylindrical firebox approximately 8.6 m long and 0.66 m in diameter, terminating at the hot face of a firetube waste-heat boiler. Combustion air was supplied to the firebox by a forced-draft fan with a capacity of 4.7 m^3/s. Most of the flue gas was vented to the atmosphere via a refractory-lined stack, 1.14 m in diameter, located above the hot face of the boiler.

The firetube boiler (2.35 MW) was designed to produce 3.76 t/h of steam at 965 kPa, although only about 50% of this capacity can normally be achieved. An induction fan drew flue gas through the boiler tubes and discharged the cooled gas into a boiler stack (0.76 m in diameter). Any flue gas not drawn through the boiler tubes was discharged through the 1.14-m diameter stack.

To serve as an afterburner, the incinerator had to be modified. The 1.14-m diameter stack has been fitted with a cap which forces all the flue gas through the boiler,

but which also serves to vent the firebox should a fuel explosion occur. The cap is in the form of a hinged captive lid that will open to relieve excessive pressure. Under normal operation, a small leak of air into the firebox occurs at this point due to the negative draft pressure. The forced-draft fan capacity was too great to allow the lower excess air levels and higher temperatures required in the afterburner, even with the control dampers closed. Therefore, the capacity of the forced-draft fan was reduced and the damper controls were reconfigured.

The induced-draft fan and its associated damper at the exit from the boiler now serve as a trim control to maintain negative pressure in the firebox rather than as a control on the fraction of flue gas drawn into the boiler. The steam produced in the boiler serves to cool the flue gas to about 315°C; the cooled flue gas is routed to the baghouse, except during a bag-shake cycle of the baghouse. Tempering air is added to the flue gas to cool it below 205°C to protect the bags in the baghouse.

The structure of the boiler has not been altered, but its service is quite different as it now accepts all of the flue gas, not a fraction of it. As a result of increased steam production and variable steam load, it is now subject to oversteaming and overheating. Because the pyrolysis gas production rate cannot respond quickly enough to avoid oversteaming at all times, a steam-dump valve and silencer have been added to the system. To conform with MOE guidelines, pyrolysis gas or solvent containing chlorine can only be burned when the firebox temperature is above 950°C, so No. 2 fuel oil is used exclusively on start-up.

3.9 Dry Scrubber

Chlorine in the fuel or pyrolysis gas burned in the afterburner is converted to hydrogen chloride; this is removed by a dry-lime scrubber, which involves the addition of dry lime to the flue gas duct downstream from the boiler exit. This lime dust is trapped with other particulate matter by the baghouse.

3.10 Baghouse

The baghouse receives all the flue gas from the boiler exit, plus the tempering air noted above. At this point, the particulate load consists of lime dust plus pyrolysis gas mineral solids (pyrolysis gas coke will have been burned). The baghouse is a Wheelabrator design with a capacity of 9.44 m^3/s (20 000 $ft.^3/min$). It is comprised of two chambers in parallel, containing 180 Nomex bags in each chamber. A fan located at the outlet of the chambers draws flue gas from the chambers and delivers it to the exit stack. The stack is

fitted with sampling ports for isokinetic gas sampling. The chambers are insulated to prevent condensation in corners where the airflow would be reduced.

As the dust loading on the bags increases, the total air drawn through them must decrease at constant fan power. This reduces the amount of tempering air because the flue gas volume is set by the forced-draft and induced-draft fans. Consequently, the temperature of the baghouse intake gas rises. When the temperature of this gas reaches the service limit of the Nomex bags, they must be shaken. This is under the control of the Programmable Logic Controller discussed below.

3.11 Control Systems

A new level of control was instituted in the demonstration plant and in the afterburner with the installation and programing of two Programmable Logic Controllers (PLCs). The volatilator section, comprising the process up to the afterburner, is now controlled by one PLC; the other PLC controls the afterburner, dry-lime scrubber, and baghouse. We chose a relatively simple model, SYMAX model 8009, because this was Anachemia's first experience with PLCs. It was felt that, under a number of circumstances, process conditions either had tc be controlled in a logical but complex way or that time did not permit leaving everything to the operator. The PLC relieves the operator of much of that burden. Under PLC control, a stoppage in one piece of equipment automatically stops or starts other equipment to minimize downtime. Indicator lights, under the control of the PLC, also show the operator the state of the process at all times.

The program for the volatilator promotes good operation by monitoring the following conditions:

- In the sand circulation train, no sand screw can operate unless the sand screw downstream from it is operating. This prevents packing sand into a stationary screw.
- Feed cannot be added unless the sand is circulating.
- Feed cannot be added unless the vapour-scrubber system is operating.

The program for the afterburner promotes good operation by monitoring or enforcing the following:

- the emergency shutdown switch
- firebox purge completion
- upper and lower firebox limits
- forced-draft fan pressure
- flame failure
- the pyrolysis gas ignition burner ON
- volatilator BLOWERS ON permission
- volatilator FEED ON permission

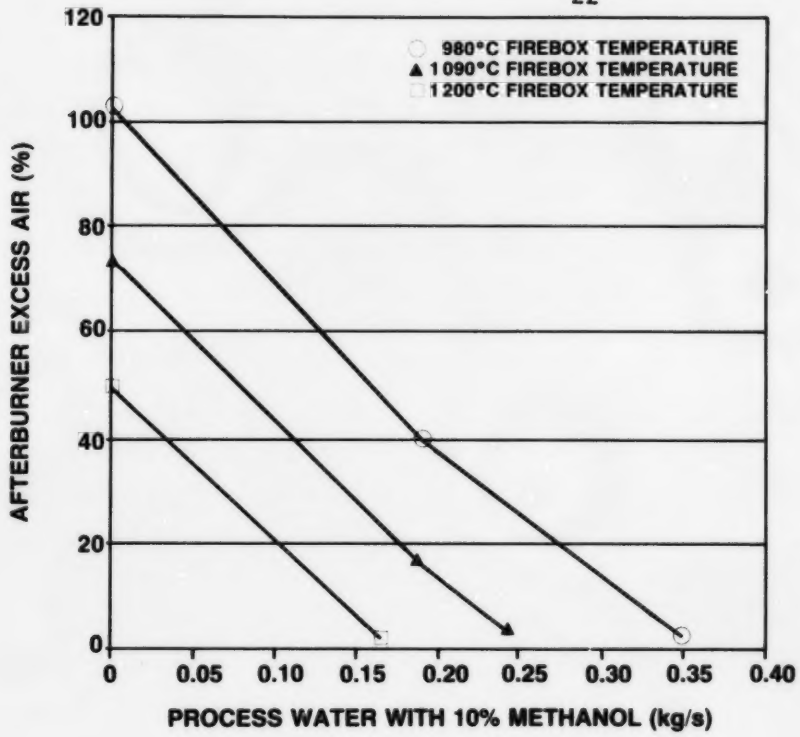
- boiler pressure limits
- the boiler water level
- baghouse operating temperature limits
- baghouse fan operation
- auxiliary fuel pump operation
- the baghouse shake cycle

These comprise most of the conditions needed to ensure safe operation. Continuously variable conditions, such as auxiliary fuel rate, are controlled by the stationary engineer.

3.12 Simulation Studies

In converting the pilot plant into the demonstration plant, substantial changes were made to the incinerator/boiler so that it would function as an acceptable afterburner. To verify the adequacy of the proposed operating conditions, the mass and heat balance calculations for the reactor, Ducon scrubber, afterburner, and waste-heat boiler were formalized into a series of simulator models to determine the relationships among steam generation, wastewater destruction, excess air, and pyrolysis gas generation rates. Each of these models (except the reactor model) must take upstream conditions from the calculated values of a particular case of its upstream model. Thus, a particular set of conditions for a boiler simulation would depend on a specific known sequence of cases for the reactor, Ducon scrubber, and afterburner. This rigid linking of models assured us that the final satisfactory results were not dependent on some unnoticed shift in boundary conditions. This also allowed us to use better methods in the calculation of physical properties, such as enthalpy, and to explore more sets of process conditions than would be practical using hand calculations. These sets of conditions, in turn, gave us valuable insight into the importance and interrelationships among process variables in the afterburner, boiler, and baghouse.

Destruction of solvent-bearing wastewater in the afterburner is an important part of our effluent-water-quality control program. However, this alters the usual relationships among excess oxygen, firebox temperature, and the residence time of gases in the firebox. Figure 9 illustrates how the excess air required to achieve a particular firebox temperature depends on the rate at which process water is destroyed in the firebox, and the effect this has on residence time in the firebox. Sufficient residence time at (or above) a minimum firebox temperature is important to ensure the destruction of dioxins and furans, should they be formed in the firebox.



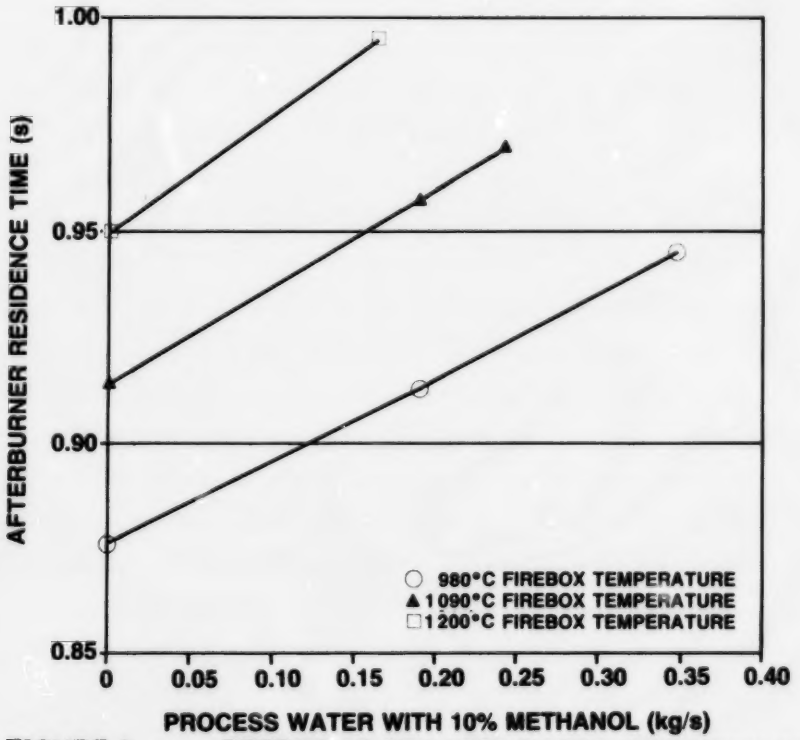


FIGURE 9 EFFECT OF PROCESS WATER INJECTION INTO THE AFTERBURNER

The results of the simulation studies are:

1). The flue-gas temperature decreased as the firebox temperature increased. The flue gas flow rate is a function of its mass and temperature. An increased excess air rate directly increases the mass flow and lowers the temperature at the hot face of the boiler. This lower temperature decreases the heat transfer (and steam production) so that the increased mass of the flue gas is also hotter. The effect of wastewater injection, generally, is to reduce the firebox temperature achieved at a given excess air rate.

2). Wastewater processing rates up to 0.4 t/h could be achieved at high firebox temperatures.

3). The MOE residence time guideline (0.75 s) for the destruction of potential dioxins and furans is met at all process water injection rates if the firebox temperature is 980°C or more.

4). For the same firebox temperature, longer residence time is achieved with higher disposal rates of wastewater. Both of these results are desirable.

To protect the bags in the baghouse, the temperature of the flue gas leaving the boiler is lowered by the addition of ambient (quench) air. However, the flow of quench air that can be used to protect the bags in the baghouse is limited to the difference between the baghouse fan capacity and the flue gas flow rate. As noted above, a higher flue gas flow rate is associated with hotter flue gas. The quench airflow, therefore, is reduced when the need for it is increased. Excess air and firebox temperature thus emerged as very important variables.

3.13 Performance

3.13.1 Run 26, December 15-23, 1986. As an example of operating performance, consider the material balance for Run 26 (Table 1) for the week of December 15-23, 1986, in which design operating rates and on-stream time were achieved. Note that certain streams (water from solvent recovery and pyrolysis gas) become internal streams and are omitted.

During a 120-h work week, with 92 h of operation, sludge was fed for 66 h at an average rate of 14.1 kg/min, producing 18 t of solvent. The afterburner was operated for 95.5 h, producing 271 t of steam and vapourizing 36 t of solvent-bearing wastewater sprayed into the firebox. (Nine tonnes were generated by the Volatilator solvent recovery, the remaining 27 t derived from other Anachemia sources.) Some of the recovered solvent was used as auxiliary fuel in the afterburner. The amount required depends on the performance level of the Volatilator. A heat balance for this run is shown

TABLE 1 MATERIAL BALANCE FOR RUN 26 (Dec. 15-23, 1986)

		Tonnes
(A) Flasher and Reactor		
Input:	Sludge processed	56
	Air to reactor	44
	Sand	2
	Fuel oil	2
	Total to reactor and flasher	104
Output:	Solvent	18
	Water	9
	Ash	7
	Pyrolysis gas	70
	Total	104
(B) Afterburner		
Input:	Pyrolysis gas	70
	Solvent as fuel	14
	Air (at 100% excess)	1697
	Lime to dry scrubber	0.1
	Solvent-bearing wastewater	36
	Total	1817
Output:	Flue gas	1815
	Ash	2
	Total	1817
(C) Combined Flasher, Reactor, and Afterburner		
Input:	Sludge processed	56
	Air to reactor	44
	Sand	2
	Fuel oil	2
	Air to afterburner (at 100% excess)	1697
	Lime to dry scrubber	0.1
	Solvent-bearing wastewater (net)	27
	Total	1828
Output:	Solvent	4
	Ash	9
	Flue gas	1815
	Total	1828

in Table 2. Note that the solvent-bearing wastewater is shown as a solvent portion (heat source) and a water portion (heat sink).

TABLE 2 HEAT BALANCE FOR RUN 26 (Dec. 15-23, 1986)

Afterburner and Boiler	Tonnes	Gigajoules
Heat Sources		
Pyrolysis gas combustibles	36	1340
Solvent fuel	14	521
Solvent in wastewater*	4	112
Total	52	1973
Heat Sinks		
Steam generation	510	1217
Flue gas	1815	560
Water in wastewater*	32	96
Losses at 5%		100
Total		1973

* Solvent-bearing wastewater comprises 4 t of solvent in 32 t of water.

3.13.2 September to March Performance Summary. Performance has been monitored from September 1986 to March 1987. Some of the data are summarized below; more detailed data are available on request.

Before taking offsetting credits, the operating cost for the period was $174 000, or $255/t of sludge processed. This includes $1000/week for maintenance, but no capital depreciation. Steam production from the afterburner boiler was 5420 t in 2400 h of operation. For approximately 40% of that time, pyrolysis gas was a contributing or major fuel; the balance of the fuel was recovered solvent. Net solvent production, nevertheless, amounted to 155 t, or 22% of the sludge feed. The net contribution to profit was $100 000.

Maintenance has been a continuing problem in the demonstration plant, although the system is not yet optimized in this regard. A total of 133 maintenance actions were recorded during the September to March monitoring period.

The solids-handling operation of the solvent-recovery plant required the most frequent maintenance. The sand-handling system required 14 maintenance actions, the cyclone system accounted for 15, preheater plugging accounted for another 2, and boiler-tube plugging (a weekly routine) added another 19 actions, for a total of 50 maintenance actions associated with solids handling. Correction of the sand circulation system requires larger diameter, slower screws and gentler slopes, which will make the

screws more efficient. The next most common maintenance (18 actions) was associated with the reactor, where the stress is due to high temperatures as well as moving sand.

Reliability: past and future. In the eight runs carried out between January 5 and March 2, 1987, about 50% of the time was spent performing maintenance. Of the operating hours logged, approximately 50% were spent processing feed. This is obviously a process that requires a lot of attention. It is felt that this performance level can still be improved substantially by upgrading the equipment.

The flue gas was discharged from the boiler at a flow rate of 66 m^3/s at about 325°C. Quench air was inducted to keep the gas going to the baghouse within the temperature limits of the bags.

4 ENVIRONMENTAL IMPACT

4.1 Introduction

The environmental impact of using this process has been to reduce solids emission by some 9 t/week, to reduce the oxygen demand of our water effluent, and to reduce the leachability of the solid waste produced as a by-product of our solvent recovery. Use of this process allows Anachemia to continue to recycle solvents recovered from paint mixtures and enhances the overall yield of recovered solvent. This avoids the burden on the environment that would otherwise be imposed as a result of the disposal of paint mixtures.

4.2 Approval by the Ontario Ministry of the Environment

The conditions of the MOE Control Order, issued on June 4, 1984, specify conformity with Ontario Regulation 309 with respect to emissions of hydrogen chloride, particulate matter, sulphur dioxide, and heavy metals, namely Pb, Cd, Hg, Ni, Cu, Zn, Mn, Cr, and Fe. In addition, Anachemia has been advised of conditions that must be met before a Certificate of Approval can be issued for the new facility. To ensure the destruction of chlorinated dioxins and furans should they be formed, it was given as a guide that the temperature of the afterburner firebox must be at least 955°C whenever fuel containing chlorine is burned in the afterburner. Also, there must be no vent to the atmosphere, except through scrubbers or dust collectors. Actual emissions must be measured under normal operating conditions and found to be in conformance before a final Certificate of Approval can be issued.

Solid wastes not emitted into the air were formerly collected and sent to municipal landfill. Under the Certificate of Approval, all solids collected are subject to the conditions of the Environmental Protection Act, and disposal at a municipal waste site is permitted only if the solids pass the leachate test discussed below.

4.3 Solids Disposal

An environmental consequence of using this process instead of simple incineration is the retention of approximately 9 t of solids per week that previously had been discharged into the atmosphere. The retained solid, if it is incorporated as such into a saleable product, is not regulated by MOE. Otherwise it must be sent to a disposal site where it will not pose a hazard to the environment. Anachemia Solvents is exploring ways to sell some of the ash as a product. However, this approach is not sufficiently developed to warrant more specific comment.

Proper disposal of ash that is not incorporated into another product depends on its properties. One test of its properties specified by MOE is the leachate test. In the leachate test, solid material, normally in granular form, is subjected to leaching by weakly acidic water. The leachate is tested for the presence of species listed in Schedule 4 of Regulation 309 of the Environmental Protection Act. If the concentration of these species exceeds the limits given in Schedule 4 by a factor of less than 10, the waste is considered to be a nonregistrable waste and may be sent to a municipal waste site. If the leachate contains these species in concentrations between 10 and 100 times the limits given in Schedule 4, it is considered to be a registrable waste and can be sent to a municipal waste site only with the permission of the municipal authorities. In all other cases, the waste must be sent to a secure landfill.

Ash produced by this process fails MOE's leachate test because the lead and cadmium ion concentrations are in excess of 100 times the allowable concentration. However, such ion species can be reacted chemically to give a less leachable form; this is termed fixing. Such "fixed" wastes may then qualify as nonregistrable wastes. The commercial fixing agent "Stablestone" has been proposed. The vendor claims that the ash, when fixed with 10% Stablestone, will pass the leachate test. Ash from our process has been treated in this way and has been found, on average, to be a nonregistrable waste. We are awaiting approval from MOE.

4.4 Air Quality

Particulate matter emissions, relative to the previous operation, have been reduced by approximately 9 t/week. With increasing pressure to allow residential development in the vicinity of the Anachemia plant and increasing numbers of people in the area in general, this reduction benefits the community directly. Lead, cadmium, and chromium are the constituents of concern; in particular, lead was found to come from one or two customers. Such customers have been asked to change their formulations to essentially lead-free recipes or their solvent-reclamation business will be rejected. No such pattern has been found with respect to chromium or cadmium.

Before MOE will issue a final Certificate of Approval, all of the requirements noted above must be certified by an independent testing laboratory. IMET (Independent Measurement and Technology) was chosen to carry out the required tests, the results of which are summarized in Table 3. We expect approval to be given by MOE in light of the results presented.

TABLE 3 FLUE GAS IMPINGEMENT CONCENTRATIONS (half-hour average)

	MOE Standards (maxima) ($\mu g/m^3$)	Anachemia Computed Test Results ($\mu g/m^3$)
Cadmium	5	2.48×10^{-4}
Mercury	5	1.90×10^{-3}
Nickel	5	2.20×10^{-3}
Lead	10	0.11
Iron (metallic)	10	0.023
Chromium	30	3.0×10^{-3}
Hydrogen chloride	100	5.5
Particulate matter	100	10.8
Copper	100	4.8×10^{-3}
Zinc	100	0.03
Manganese	100	5.2×10^{-4}
Sulphur dioxide	830	70

4.5 Water Quality

The solvent-recovery operation, which is the principal business of Anachemia Solvents, deals with solvent mixtures that contain water. Some of this water separates as a water phase containing common solvents. If released to the municipal drains, the solvent content would constitute an oxygen demand in the municipal wastewater treatment plant. Such oxygen demand is subject to a surcharge by the municipality and is further limited by a municipal bylaw. Anachemia reduces the amount of solvent discharged to sewer by additional phase separation, solvent stripping operations, and "burning" some of the solvent-containing water in the afterburner as outlined previously. The afterburner is able to process approximately 9 t/d in this fashion. This improves the water quality of our effluent and reduces the surcharge.

5 ECONOMICS AND ENERGY SAVINGS

In the six months ending April 6, 1987, the process contributed some $100 000 to profit, of which $40 000 is the estimated value of net solvent production. Steam production amounted to 5400 t, which is equivalent to a saving of about 340 000 m^3 of natural gas. In addition, the uncertainty of shipping sludge to a secure landfill is reduced, which allows additional business to be accepted. This potential opportunity profit is not included.

Charges made to Anachemia's customers must reflect both the solvent-recovery cost and the attendant disposal cost of waste generated. An improvement in the disposal cost, therefore, helps Anachemia's customers remain competitive.

The effect on the economic balances of the country are, of course, quite minor in the context of total trade. Nevertheless, the directional effects can be discerned locally. Still-bottom sludge that must be shipped out of the country for disposal represents money that leaves the country. Solvent that is recycled, on the other hand, represents an equivalent amount of crude oil that need not be imported or that becomes available for export from Canada. Operation of this process, therefore, is beneficial to Canada.

6 CONCLUSIONS AND FURTHER WORK

The process reported here is a technical and economic success. The hot-sand technique is able to separate solvent from the feed material without turning the residue into a solid mass. The process supplies the heat energy to maintain itself and the pyrolysis gas provides fuel to a waste-heat boiler. The solids content of the feed is recovered as a fine powder by cyclones and by a baghouse for disposal in an environmentally acceptable manner. The economic effect of the process is positive due to the reduction in sludge disposal costs, lower steam production costs, and the ability to dispose of solvent-rich process water from the conventional solvent-recovery operations on-site.

There is room for improvement, however, in the reliability and process capacity of the present unit. Indeed, the next configuration is under construction. It will be characterized by a better layout, greater efficiency of the sand screws through a reduction in the change in elevation of the circulating sand, and better facilities to move solvent, sand, and process water.

This process could also be developed to treat other slurries of combustible liquids and mineral solids, e.g., oil-well drilling muds.

REFERENCES

1. Fane, A.G. and R.A. Mitchell, "Minimum Spouting Velocity of Scaled-up Beds," Canadian Journal of Chemical Engineering, 62:437 (1984).

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